ANALYTICAL CHEMISTRY DATA MANAGEMENT AND REVIEW FOR RAD-NESHAP PROGRAM

Purpose

This Meteorology and Air Quality Group (MAQ) procedure describes the process for receiving, uploading, and archiving both field sampling and analytical chemistry data from the NESHAP compliance project; evaluating analytical chemistry quality; checking the resulting chemistry data packages for completeness and usability; and conducting validation/verification of both electronic and hardcopy data from both current and historical (pre-1997) sources.

Scope

This procedure applies to all analytical chemistry needs of the MAQ Rad-NESHAP project.

In this procedure

Signatures

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General information about this procedure

Attachments

This procedure has the following attachments:

Number	Attachment Title	No. of pages
1	QC Evaluation Criteria	1
2	General Completeness of Data Packages for NESHAP program	1

History of revision

This table lists the revision history and effective dates of this procedure.

Revision	Date	Description of Changes
0	5/9/01	New document.
1	04/14/06	Changed alpha spike and duplicate requirements to address deficiencies 450 and 498. Updated General Completeness Checklist.

Who requires training to this procedure?

The following MAQ personnel require training before implementing this procedure:

- NESHAP Field Team
- Analytical chemistry data reviewers
- Analytical Chemistry Coordinator/NESHAP Data Manager

Training method

The initial training method for this procedure is **mentored** training by a previously trained individual, and is documented in accordance with the procedure for training (MAQ-024).

Annual retraining is required and will be by self-study ("reading") training.

Prerequisites

In addition to training to this procedure, the following training is also recommended prior to performing this procedure:

- Education and/or experience in compliance-oriented analytical chemistry
- Familiarity with Microsoft Access
- Familiarity with the operation of the RADAIR database

General information, continued

Definitions specific to this procedure

Statement of Work (SOW): A list of specifications and requirements which analytical laboratories must meet in order to do work for MAQ.

Data Package: A hardcopy report from an analytical laboratory on a single set of chemical analyses, which contains the material specified in the SOW and sufficient documentation to allow an appropriate professional, at a substantially different time and location, to ascertain:

- what analyses were performed, and what results were obtained
- that the data had acceptable properties (such as accuracy, precision, MDA)
- where, when, and by whom the analyses were performed
- that the analyses were done under acceptable conditions (such as calibration, control, custody, using approved procedures, and following generally approved good practices)
- that the MAQ SOW was otherwise followed.

Completeness: A measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under ideal conditions.

Usability: A qualitative decision process whereby the decision makers evaluate the achievement of data quality objectives and determine whether the data may be used for the intended purpose. Three levels or classes of data quality are used:

- Accepted: Data conform to all requirements, all quality control criteria are met, methods were followed, and documentation is complete.
- Qualified: Data conform to most, but not all, requirements, critical QC criteria are met, methods were followed or had only minor deviations, and critical documentation is complete.
- Rejected: Data do not conform to some or all requirements, critical QC criteria are not met, methods were not followed or had significant deviations, and critical documentation is missing or incomplete.

Electronic Data Deliverable (EDD): The computer-compatible file that is delivered to MAQ from the analytical laboratory, in the SOW-specified format, via Internet, e-mail, or diskette from which analytical chemistry data may be uploaded directly into the databases.

General information, continued

Definitions specific to this procedure, *continued*

Validation: A systematic process for reviewing a body of data or a report against a set of criteria to provide assurance that the data or report are adequate for their intended use. Validation consists of data reviewing, screening, checking, auditing, verification, certification, and review.

Verification: The act of reviewing, inspecting, testing, checking, auditing, or otherwise determining and documenting whether items, processes, services or documents conform to specified requirements.

References

The following documents are referenced in this procedure:

- MAQ-024, "Personnel Training"
- MAQ-026, "Deficiency Reporting and Correcting"
- MAQ-036, "Preparing Statements of Work for Procuring Analytical Chemistry"
- MAQ-039, "Web Page Posting and Maintenance"
- MAQ-106, "Collecting Tritium Stack Bubbler Samples"
- MAQ-109, "Collecting Stack Particulate Filter and Charcoal Cartridge Samples"
- MAQ-135, "Collecting Beryllium Stack Filter Samples"
- MAQ-601, "Collecting and Processing Stack Air Particulate and Vapor Samples from TA-53"
- MAQ-RN, "QA Project Plan for the Rad-NESHAP Compliance Project"
- RADAIR Database Users Guide

Note

Actions specified within this procedure, unless preceded with "should" or "may," are to be considered mandatory guidance (i.e., "shall").

Background

Description of process

Stack monitoring is conducted by the MAQ Rad-NESHAP Project team to demonstrate compliance with the Clean Air Act (40 CFR 61, Subpart H), using the provisions incorporated into this federal law or the Federal Facilities Compliance Agreement of 1996 between the EPA and the DOE that details how certain provisions of the Act would be applied to the Laboratory.

To facilitate understanding this procedure, it is worthwhile to note how the sampling portion of the Project is structured. Stacks are organized into three groups: particulate, tritium, and LANSCE. Glass fiber filter samples are taken in all particulate and LANSCE stacks and in-line charcoal canisters are included after the filter in a subset of those emission points. Bubbler samples are taken in the tritium stacks to monitor HT and HTO gas emissions. The variety of locations, emission types, sampling media and isotopes of concern provide for complex analytical and data management needs to support this critical compliance program.

Requirements for chemical analyses are described in the data quality objectives (DQO) sections of the several Quality Assurance Project Plans for which the samples are collected. Data quality objectives from these quality assurance project plans are translated into procurement needs and related Statements of Work (SOW) according to MAQ-036. Field data are taken manually by the NESHAP field sampling team at the time the sampling media are changed. Samples are hand-carried to internal chemistry laboratories or shipped via overnight carrier to external commercial suppliers. Field data are manually entered into the RADAIR database by the field team immediately after collection and then archived to limited-access tables to protect their integrity. Sample analysis data are first received in an electronic format (EDD) from all internal and external analytical chemistry sources under these SOWs, uploaded electronically into the RADAIR database, archived to limited-access tables to protect their integrity, and then released to facility personnel in preliminary form via the MAQ Internal web page. Approximately 1-2 weeks later a formal, hard-copy data package is received and both data package and electronically uploaded data are inspected to determine if they meet MAQ specifications. This inspection, using checklists prepared by the analytical chemistry coordinator from the SOW, includes checking the data package received from the laboratory to ensure that:

- the data package contains the components specified in statements of work,
- all of the requested analyses were performed for all samples,
- the data are of a quality adequate for the use which MAQ intended, and
- all data received electronically are verified against those in the hard-copy data package to ensure agreement.

Background, continued

Continued

All manually entered data and only a portion of the electronic data (usually 10%) are verified against the hard copy to ensure exact reproduction of the analytical concentrations, and the data usability are evaluated for acceptance, qualification, or rejection.

For RADAIR, initial emissions values and evaluation against the 0.01 mrem dose trigger are sent to the project health physicist, along with summaries of all analytical QC data. When documented data review and proposed actions are received back from the health physicist, these actions are posted to the RADAIR database. Corrected data are re-posted to the MAQ internal web page. All stages of the process are tracked electronically within the database.

MS Access RADAIR Data base overview

A database has been designed and implemented in Microsoft Access that is specific to the needs of the Rad-NESHAP project. This application is form-driven, with all parts of the process accessible from a Main Switchboard form. Each sub-form provides a series of labeled buttons presented in correct order to facilitate the easy implementation of any of the data management processes needed to support this project. Users Guide information is provided as electronic media that can be accessed directly by “Help” buttons located on each form.

Preparing checklists for deliverables

When to prepare completeness checklist

The **MAQ analytical chemistry coordinator** prepares checklists as needed to evaluate the completeness of any deliverables when new services are procured. Base the checklists on the SOWs, EDDs, electronic database designs, and professional judgment. Tailor the checklist formats to allow easy checking of analyses purchased frequently (such as weekly analyses for gross alpha/beta and gamma-emitting isotopes or beryllium, and semiannual composites analyses for alpha, beta, and gamma emitting isotopes). As such, the sequence components may be different in the checklist and SOW, but all content is to be included. Current versions of these checklists are available directly from one or more of the RADAIR database forms.

Examples of current checklists are attached to this procedure as Attachments 2 and 3.

Steps to prepare a checklist

Follow these steps to prepare checklists:

Step	Action
1	Consult the relevant SOW, EDD, and RADAIR database table design specifications to identify the supporting documentation required.
2	Consult an existing checklist, if available, matching requirements as closely as possible.
3	Obtain a sample data package for the analyses from the lab.
4	Prepare the new data package completeness checklist by modifying an existing checklist to match current requirements and package sequence. Ensure the data reviewers have the current versions.
5	Prepare the new database completeness and V&V checklist by modifying an existing checklist to match current structure of the RADAIR database. Ensure the data reviewers have the current versions by posting it to the MS Access Form from which this asset is called.

Entering RADAIR field sampling data

Purpose of upload

Currently all field sampling data for this program are manually entered on paper forms from procedures MAQ-106, -109, -135, and –601. These data are entered into the database to make them readily available to all NESHAP program staff and supporting software applications, and provides for the use of automated means to evaluate the quality, completeness and representativeness of these data.

Steps to upload field sampling data

Manual data entry into the MS Access RADAIR database is conducted by the field sampling team using various MS Access Forms provided within the database and documented in detail in the RADAIR Database Users Guide. Each stack group (particulate, tritium, and LANSCE) has different field data parameters, necessitating special software for each group.

Step	Action
1	Collect recent original field sampling data sheets from procedures MAQ-106, -109, -135, and -601 within three days of the end of each weekly sampling period. Obtain access to a computer terminal connected to the MAQ group server.
2	Log-in to the RADAIR Database. The Main Switchboard form is automatically displayed. Open the sub-form that is specific to the type of field data being entered. Complete the data entry process specific to each stack group documented in detail in the RADAIR Database Users Guide.
3	Archive these field data into limited access tables within the RADAIR database using the process documented in detail in the RADAIR Database Users Guide.
4	Have a second person verify and validate 100% of these manually entered field data immediately after uploading to the RADAIR database.
5	Document the completion of all phases of the field data handling process using the field data tracking software in the RADAIR database.

Processing and evaluating the EDD for RADAIR analytical chemistry data

Electronic data deliverables

EDDs may be received from both internal and external analytical chemistry laboratories. Format and content requirements are specified in each individual Statement of Work prepared according to MAQ-036. Each EDD requires specific software to enable them to be incorporated into the existing databases. The uploading process is facilitated by using the form-driven software application RADAIR, and is described in detail in the RADAIR Database Users Guide.

Steps to upload EDD

To upload and evaluate incoming EDDs, follow the steps below:

Step	Action
1	Upload EDDs: As soon as practical after receipt, upload EDDs by following the appropriate steps in the RADAIR database menus. Use of the database is described in detail in the RADAIR Database Users Guide.
2	Evaluate against SOW requirements: After uploading data received electronically, inspect the data visually just prior to its transfer to the archive table. Evaluate this deliverable to ensure that all components are the same as those usually received or required by the SOW and that it has not become corrupted during the transmission process.
3	If any required data components are missing or errors detected, contact the lab immediately and request that a revised EDD be sent expeditiously.
4	Archive data: Follow the database menu steps to archive the data for further review.
5	Notify the analytical chemistry coordinator that the data have been uploaded.
6	Notify the individual who is responsible for releasing the preliminary data via the WWW (see procedure MAQ-039) to facility personnel. **NOTE**: the data at this point are still subject to change after further review, as described in the remainder of this procedure.

Processing and evaluating the EDD for RADAIR analytical chemistry data, continued

Custody errors

Custody errors are those which make it difficult to demonstrate that the samples that were shipped by MAQ were the same as those analyzed by the lab. Examples include:

- MAQ or lab staff not signing and dating chain of custody forms
- Loss or miscounting by MAQ or the lab
- Misidentifying by MAQ or the lab
- Lost samples
- Delivery to the wrong site or person

Document any custody errors with an MAQ Deficiency Report (MAQ-026). Resolution will require coordination with the lab. If new analyses are necessary, ship the new samples under a new chain of custody.

Check hard-copy of data package

The hard-copy of data packages are usually received a week or more after the EDD. After receiving the hard copy, follow the steps below to check the data package.

Step	Action
1	After receiving the final hard-copy data package, print the V&V checklists (Attachment 2 and 3).
2	Print the chemistry data to be checked (for gamma data, these are normally the Co-60 and Cs-137 results). This complies with the requirement to check 10% of electronically loaded data.
3	Use the appropriate checklist to evaluate the deliverable and compare the printout to the hard-copy package. If there are any discrepancies, contact the lab immediately.
4	After correcting any problems and/or entering comments in the database, sign the printout and the checklists.
5	Record V & V completion of all phases of data upload using the appropriate sample tracking software options in the RADAIR database.
6	Use the appropriate menu options to print the data reports for all data package types.
7	Using the appropriate database menu options, open the internal QC memo.
8	Evaluate the data against the limits in the memo and reports.

Steps continued on next page.

Processing and evaluating the EDD for RADAIR analytical chemistry data, continued

Step	Action
9	In the memo and attached reports, edit appropriate fields for the data package reviewed. On page 2, edit or enter appropriate information regarding the evaluation and enter any comments on each review item.
10	Print the internal QC memo, initial it, and forward to the analytical chemistry coordinator or health physicist for technical review.

Analytical chemistry data evaluation

The data evaluation by the analytical chemistry coordinator determines whether chemical analyses data meet the data quality objectives specified in the quality plan (e.g., MAQ-RN). All data will be evaluated for one of three outcomes: *accept*, *qualify*, or *reject*. For qualified and rejected data, an explanation must be included in the database.

The **analytical chemistry coordinator** reviews the internal QC memo and the attached reports and further evaluates the data against the criteria in Attachment 1. The signature of the analytical chemistry coordinator indicates that the data meet the listed criteria. Forward the package to the health physicist for review, if this has not been done already.

Health physicist review

The **health physicist** responsible for routine review of these data reviews the internal QC memo and the attached reports and further evaluates the data against the criteria in Attachment 1. The signature of the health physicist indicates that the data meet the listed criteria. Forward the package to the analytical chemistry coordinator for review, if this has not been done already.

HP action implementation

After the project health physicist and analytical chemistry coordinator conduct their reviews, follow the steps below to implement changes in acceptance outcomes in the archive tables within the RADAIR database.

Step	Action
1	If applicable, implement the recommended actions in the database and document the reasons in the comment field.
2	If any changes to preliminarily reported data have been made, republish emissions tables and plots to the MAQ Internal web page.
3	Ensure the fully approved summary data are published to the MAQ WWW homepage according to procedure MAQ-039.

Evaluation of RADAIR pre-1997 field and analytical data

Purpose of data evaluation

Data collected prior to 1997 were not procured to the same standards, did not have the same data package documentation, and cannot be reviewed to the same level as 1997 and subsequent data. As part of an on-going process, these data are being reviewed to the extent practical and made available electronically in the RADAIR database. Since data are being loaded from a variety of sources using both electronic and manual means, all data must undergo verification and validation to ensure the correctness of the electronic record.

Steps to evaluate data

Perform the following steps to evaluate field sampling and analytical chemistry data:

Step	Action
1	Collect available hard-copy field sampling and analytical chemistry data records for the sampling period being evaluated. Obtain access to a computer terminal connected to the MAQ group server.
2	Evaluate for completeness to the extent permitted by the existing records. Each field or analytical data element should have a value. Ensure an explanation is recorded in the database for all missing data. • If a missing datum is without an acceptable explanation, attempt to determine the reason, label the datum "qualified" in the database and enter the reason for qualification. • If unable to determine a reason, leave the field blank and enter "R" in the qualifier field.
3	Evaluate for expected range of values, to the extent permitted by the existing records. For example, the expected range might be a nominal value with a range of possible values. Project quality plans often list some of the expected values for data elements.
4	As a result of step 3, if the element is outside its range of normal values or some field event renders the data potentially suspect, identify the record as "qualified." Perform further validation and verification by consulting with the field sampling technicians to determine what conditions at a site may have resulted in the data value reported. Label any amended field records as "qualified" (enter a "Q" in one of the field data qualification fields) and describe in the table's comment field the amendments made.
5	If the data were not used in prior year's calculations or reports, label the data record as "rejected" (enter an "R" in one of the field data qualification fields) and provide the reasons for rejection in table's comment field.

Steps continued on next page.

Evaluation of RADAIR pre-1997 field and analytical data, continued

Step	Action
6	Move the data to the archive tables within the RADAIR database for use in published reports and for release to the public. Specific procedures are documented in the RADAIR Database Users Guide.

Records resulting from this procedure

Records

The following records generated as a result of this procedure are to be submitted **within 3 weeks of their receipt or generation** as records to the records coordinator:

- RADAIR Completeness of Data Package (SOW-01) form; completed, signed, and dated
- RADAIR Field Data Validation and Verification Database inspection memo; completed, signed and dated.
- RADAIR Analytical Data Validation and Verification Database Inspection form; completed, signed and dated.
- Copy of final laboratory data packages
- Deficiency reports resulting from chain-of-custody problems, if any
- MAQ internal memos documenting data quality evaluation, data validation, and initial air emissions calculations

The following electronic records generated as a result of this procedure are to be contained within their respective Microsoft Access databases:

- entries in RADAIR databases for all accepted, qualified and rejected data from both field and analytical processes.

QC EVALUATION CRITERIA

Type of Data	Evaluation Performed	Acceptance Criteria
All	Laboratory Control Standard (LCS) recovery check	100 ± 20% for gross alpha/beta and 100 ± 10 % for all others.
All except Alpha/Beta	Process Blank (PB)	See Control Criteria below
All	Matrix Blank (MB)	See Control Criteria below
All	Trip Blank (TB)	See Control Criteria below
Be	Matrix Replicate evaluation	See Control Criteria below
Alpha/Beta, alpha and beta isotopics	Matrix Replicate evaluation	Duplicate Error Ratio less than 2 (in control). The formula for the DER is given below.
H-3	Matrix Duplicate evaluation	Calculate the RPD for each duplicate generated by the analytical laboratory using the standard EPA formula. Evaluate by concentration level against historical analytical laboratory performance
Gamma	Matrix Replicate evaluation	Duplicate Error Ratio less than 2 (in control). The formula for the DER is given below.
H-3 *	Matrix Spike	100 ± 10% of added spike
All	MDA achieved	All samples below SOW specification
All	Missing Field or Analytical data	No missing data for actual field samples
Gamma	Unknown isotopes	Note energy of unknowns in database and make reasonable attempt to identify them
Each weekly period	Sampling Station Run Time completeness	95% up-time
All	Analytical Completeness	80% successful analysis of valid samples
All	Dose Action Level Comparison	< 100% of target value

General Control criteria:
"Under control" is within <= 2s of annual mean for that QC type
"Warning" is between 2s and 3s of annual mean for that QC type
"Out of control" is >= 3s of annual mean for that QC type

Duplicate Error Ratio

The Duplicate Error Ratio is the comparison of the difference between the sample and replicate results to the propagated error values. It is calculated as follows.

$$DER = 2 * \frac{|S - R|}{\sqrt{(2s_S)^2 + (2s_R)^2}}$$

where:

DER = Duplicate Error Ratio between a sample & its replicate
S = Result of the original sample
R = Result of the replicate analysis
$2s_S$ = Two-sigma error of the original sample
$2s_R$ = Two-sigma error of the replicate analysis

"In Control" if the DER is less than 2.0
"Warning" level if the DER is between 2.0 and 3.0
"Out of Control" if the DER is greater than 3.0

Note: most DER's are analyzed with one-sigma ratios, but with control and warning levels adjusted appropriately to achieve the same goal.

* A matrix spike is not performed for alpha, beta, or gamma analysis. These spike samples were prepared by the analytical laboratory in a manner almost identical to the preparation of the LCS. In order to avoid redundant analysis, the matrix spike samples were omitted for all analytes except tritium. The tritium spike samples are prepared by MAQ personnel and sent in for analysis with the field samples.

General Completeness Of Data Packages for NESHAP Program

Form Version: 09/21/2005

RADAIR Sample Number: _____

Analysis Type:	**Sample Type:**
Alpha/Beta Gamma Tritium Beryllium Alpha Isotopics	**Bubbler Charcoal Filter Paper**

Inspection Criteria	**Criterion Met, Not Met, N/A**	**Comment?**
ALL DATA PACKAGES		
Narrative comments in cover letter or memo	Y N NA	´
Positive sample id in all tables and reports/ cross check LANL and Lab ID	Y N NA	´
Copy of the Chain of Custody form/ Submittal form(for tritium)	Y N NA	´
Data received for each sample on Chain of Custody/ Submittal form(for tritium)	Y N NA	´
Information in body of data package: • Preparation and analysis method • Isotope or analyte • Analyte concentration • Analyte uncertainty (noted as two sigma) • Analysis MDA • Dates and time of analysis • Consistent units (result, uncertainty, MDA) • Appropriate comments, notes, qualifiers	Y N NA	´
Laboratory QA/QC samples One of each for every 20 field samples: • Laboratory Control Sample (LCS) • Duplicate analysis • Laboratory-provided matrix blank *When available, the following blanks appear:* • *LANL-provided matrix blank* • *Trip blank* • *Spike blank*	Y N NA	´
Known values for all QA/QC samples	Y N NA	´
Individual sample and QC raw data	Y N NA	´
Individual detector efficiencies and backgrounds.	Y N NA	´
Laboratory bench sheets and calibration records	Y N NA	´
Evidence of traceability for calibration standards	Y N NA	´
Alpha/Beta only		
Analysis dates in memo, Load Order Sheet and individual Analytical Reports ALL match	Y N NA	´
Blank corrections made to data	Y N NA	´
Gamma only		
Individual sample and QA/QC sample raw data and individual spectral plots showing regions of interest (ROI) integrated for each isotope.	Y N NA	´
Alpha Isotopics only		
Tracer activity	Y N NA	´
Tracer recovery will be reported as fractional percent	Y N NA	´
Tritium in Glycol only		
Spike data	Y N NA	´
Actual pipette volume used in tritium calculations	Y N NA	´
Instrument performance charts for background, efficiency, figure of merit, Chi-Square , tSIE tables, in the raw data	Y N NA	´
Beryllium on Filters only		
Filter halves analyzed and reported	Y N NA	´
Data V&V method used		
10% (100% for Be) of EDD	Y N NA	´
All dates filled in on tracking table	Y N NA	´
Comments:		

Verified by: _____Date: _____